



Original research article

Study on Raman spectra of two expansins in pure solid-phase and in water solutions



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ABSTRACT

The Raman spectra of two kinds of expansins KT-30 and DA-6 are measured and analyzed in the cases of pure solid-phase and in water solutions. The results show that, KT-30 has much richer and narrower Raman spectral peaks. The first, second, and third strong Raman peaks are respectively located 990.68738 cm^{-1} , 1606.0975 cm^{-1} , and 1327.9379 cm^{-1} . Besides the dense and narrow Raman peaks below 1800 cm^{-1} , there exist three weak peaks within $3050\text{--}3410\text{ cm}^{-1}$. In comparison, fewer spectral peaks can be observed for DA-6. The first and second strong peaks respectively appear at $2883.03760\text{ cm}^{-1}$ and 2848.4480 cm^{-1} . There exist about 8 peaks below 1500 cm^{-1} . In water solutions, bell-shaped and homogeneous fluorescence background will respectively superpose on the Raman spectra of KT-30 and DA-6 within $100\text{--}2500\text{ cm}^{-1}$ and $400\text{--}4000\text{ cm}^{-1}$. Parts of spectral peaks will be covered accordingly. Moreover, the peak position shifts of KT-30 are very slight. While DA-6 takes the opposite. Its largest shift can even be up to 63.4148 cm^{-1} . The results can supply important fingerprint data for Raman detection of expansins in fruits and vegetables.

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1. Introduction

It is really very important especially for agricultural country China to pay attention to high yielding of fruits and vegetables and developing agriculture through science and technology. However, safety of agriculture products is obviously much more important anytime in any country. As important plant growth-promoting agents, expansions can be appropriately utilized to stimulate the cell division and speed up the growth of fruits and vegetables in order to achieve the object of acquiring high yielding. However, overuse of expansins may lead to various harmful negative effects such as fruit variation, hollow fruits, bursting melon, poor palate, and nutrition reduction [1,2], etc. Furthermore, it may be harmful to people's health to eat the fruits and vegetables with high residues of plant growth-promoting agents for long [1,2]. For example, it may cause early development or precocious puberty in children. And what's worse is that it may even hurt human important organs. Therefore, it is of great importance in ensurance of quality and product yields of fruits and vegetables, to detect efficiently, rapidly, and non-destructively, the residues of expansins in fruits and vegetables. So far, chromatographic methods are mainly utilized to detect the residues of expansins in fruits and vegetables [1–3]. However, chromatographic methods are time consuming and complex in sample procedure. In comparison, Raman spectrum method has advantages of high efficiency, damage-free, rapidity, simplicity in sample procedure, and etc. Up to now, there are reports on analysis of food hygiene

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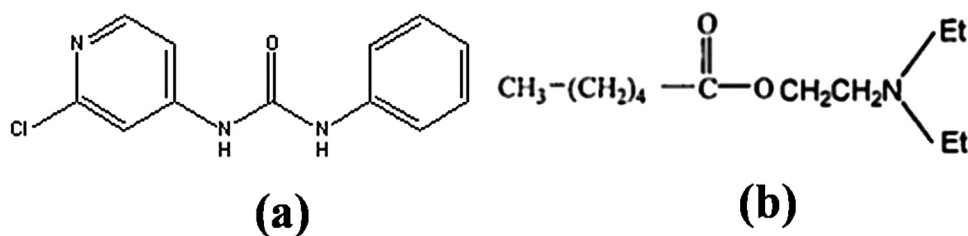


Fig. 1. The structural formula of KT-30 (a) and DA-6 (b).

and safety by Raman spectroscopy. For instance, utilizing Raman spectroscopy to analyze the changes of arabinoxylan in wheat endosperm cell walls during grain filling [4], to study the deamidated food proteins [5], to determine the amylose content in starch [6], to investigate the mechanism of thermal activated radical formation in potato starch by combining the electron paramagnetic resonance method [7], to detect the pesticides residues in fruits and vegetables [8–10], and so on. In particular, recent developed surface-enhanced Raman spectroscopy technology raises the detection sensitivity considerably [11–13]. However, to our best knowledge, reports on detection of expansin residues in fruits and vegetables are rarely seen [13]. In Ref. [13], KT-30 and TDZ residues in fruits and vegetables are analyzed by combining the surface-enhanced Raman spectroscopy and selective solid-phase extraction [13]. However, its Raman spectra are limited to the frequency shift range 500–2000 cm^{-1} .

When one detects the residues of KT-30 and DA-6 in fruits and vegetables by Raman spectroscopy, the corresponding fingerprint Raman spectra must be obtained in advance. Based on these ideas, the fingerprint Raman spectra of KT-30 and DA-6 are experimentally measured within the range of 100–4000 cm^{-1} in pure solid-phase and water solution in this work, which can serve as important data sources for Raman detection of expansins in fruits and vegetables.

2. Experiments and discussions

The molecular formulas of DA-6 and KT-30 are respectively $\text{C}_{12}\text{H}_{25}\text{NO}_2$ and $\text{C}_{12}\text{H}_{10}\text{ClN}_3\text{O}$. DA-6 and KT-30 are respectively bought from Zhengzhou Runyu Agricultural Science and Technology Co., Ltd, Henan province, China, and Xinyu Chemical Science and Technology Co., Ltd, Henan province, China. Their purities are 98% and 99% respectively. And their structural formulas are shown in Fig. 1.

Distilled water is used as solvent. The masses of DA-6 and KT-30 are weighted by using electronic balance. For the reason that KT-30 is poorly soluble in water, it is prepared as saturable solution. While the concentration of DA-6 in water is prepared to be 32.6%. LabRAM HR typed Raman spectrometer made in HORIBA Co., French, is used in our experiment to collect Raman spectra. The corresponding excitation wavelength is 532.17 nm. The laser power irradiating on the sample is mW level.

The obtained Raman spectra of DA-6 in pure solid-phase and in water solution are shown in Fig. 2a and b respectively. In the case of pure solid-phase, one can see from Fig. 2a that, the first and second strong peaks are next to each other and respectively appear at 2883.03760 cm^{-1} and 2848.4480 cm^{-1} . In the range of 1000–1500 cm^{-1} , there exist about eight moderate Raman peaks. In the whole range 100–4000 cm^{-1} , there are about thirteen peaks in all. From small to large frequency shifts, the corresponding positions of Raman peaks are 140.35477, 418.5144, 597.22839, 836.47449, 889.80042, 1064.1907, 1085.8093, 1129.0465, 1297.6719, 1436.03, 2725.9424, 2848.448, and 2883.0376 cm^{-1} successively. We have marked them in Fig. 2a.

Raman spectrum of DA-6 will vary in water solution. As shown in Fig. 2b, owing to influence of water solution environment, an obvious change is that the intense and homogeneous fluorescence background exists in the spectrum. Moreover, in comparison with the pure solid-phase case, the two intense peaks are also next to each other but their positions respectively shift to 2946.4524 cm^{-1} and 2900.3325 cm^{-1} . There are about ten Raman peaks altogether in the whole range 100–4000 cm^{-1} and they are successively located 447.33923, 517.96008, 564.07983, 850.8869, 905.65411, 1116.0754, 1120.3992, 1454.7672, 2900.3325, and 2946.4524 cm^{-1} .

In order to compare the spectra of DA-6 in pure solid-phase and in water solution, we put them together in the same figure, as shown in Fig. 2c. It can be seen that, some important spectral peaks correspond with each other but with some red or blue shifts. Concretely, the Raman peaks 418.5144, 597.22839, 836.47449, 889.80042, 1064.1907, 1129.0465, 1436.031, 2848.448, and 2883.0376 cm^{-1} , in pure solid-phase, respectively correspond to 447.33923, 564.07983, 850.8869, 905.65411, 1116.0754, 1120.3992, 1454.7672, 2900.3325, and 2946.4524 cm^{-1} in the water solution. The smallest and largest position shifts are respectively 8.6473 and 63.4148 cm^{-1} . Accordingly, owing to influence of water, besides occurrence of intense fluorescence background on the Raman spectrum, the Raman peaks may exhibit large red or blue frequency shifts. Moreover, some spectral peaks are covered by the fluorescence background and cannot be seen any longer.

The Raman spectra of KT-30 in pure solid-phase and in water solution are shown in Fig. 3a and b respectively. In the case of pure solid-phase, one can see from Fig. 3a that, the spectral peaks are very rich. Especially, there are about thirty narrow peaks with different intensities in the range of 100–1800 cm^{-1} . The first, second, and third strong peaks are respectively located 990.68738, 1606.0975, and 1327.9379 cm^{-1} . In the large frequency shift

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